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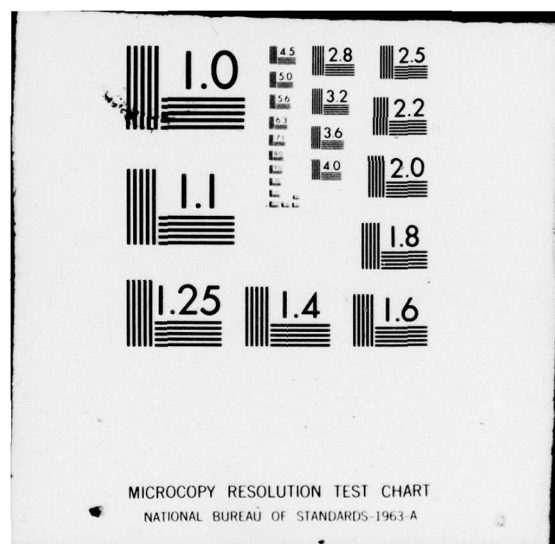
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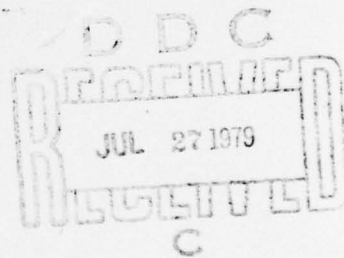
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Report 2276

DETERMINATION OF BRANCHED CHAIN KETONES
IN COATING SOLVENTS

by
Troy Nichols
George Svarnas
and
Robert E. Thomas

May 1979



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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 2276	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER 9 Rest. for
4. TITLE (and Subtitle) 6 DETERMINATION OF BRANCHED CHAIN KETONES IN COATING SOLVENTS		5. TYPE OF REPORT & PERIOD COVERED Apr 1978 - March 1979
7. AUTHOR(s) 10 Troy Nichols George Svarnas Robert E. Thomas		6. PERFORMING ORG. REPORT NUMBER
8. PERFORMING ORGANIZATION NAME AND ADDRESS Material Technology Laboratory U.S. Army Mobility Equipment Research and Development Command; Fort Belvoir, Virginia 22060		8. CONTRACT OR GRANT NUMBER(s)
9. CONTROLLING OFFICE NAME AND ADDRESS Material Technology Laboratory, DRDME-V U.S. Army Mobility Equipment Research and Development Command; Fort Belvoir, Virginia 22060		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS PRON No. A1-8-P635-01-AW-EF
11. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 12 21 p.		11. REPORT DATE 11 May 1979
12. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		12. NUMBER OF PAGES 23
13. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) 14 MERADCOM-2276		13. SECURITY CLASS. (of this report) Unclassified
14. SUPPLEMENTARY NOTES		14a. DECLASSIFICATION/DOWNGRADING SCHEDULE
15. KEY WORDS (Continue on reverse side if necessary and identify by block number) Branched chain ketones Air pollutants Coatings solvents GLC analysis		
16. ABSTRACT (Continue on reverse side if necessary and identify by block number) → This report covers the development of and results obtained with an analytical procedure to detect and determine the branched chain ketones: methyl isobutyl ketone (MIBK), methyl isoamyl ketone (MIAK), mesityl oxide (MO), and isophorone in paint solvents. Two spot tests, a 2,4-dinitrophenylhydrazine separation and a quantitative gas-liquid chromatographic (GLC) technique, are used. ← 403 260		

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PREFACE

The work covered by this report was performed by Troy Nichols, George Svarnas, and Robert E. Thomas and was reviewed by Martha L. Adams under the supervision of Emil J. York, Chief, Material Technology Laboratory. Debra LaVine assisted in the development of data for this report.

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DETERMINATION OF BRANCHED CHAIN KETONES IN COATING SOLVENTS

I. INTRODUCTION

1. **Subject.** The object of this investigation was to develop a method for the determination of branched chain ketones in organic coating solvents in order that these materials may be used to the extent permitted by legislation.

2. **Background.** Ketones, both linear and branched chain, have been one of the more effective classes of materials for difficult solubility problems in organic coatings. Present emphasis, however, is toward restriction of the use of *branched* chain ketones because of their photochemical reactivity. Their use in Military Specifications for organic coatings is limited by Composition L requirements (Table 1), which are based on Rule 442 (formerly Rule 66) of California.

Table 1. Composition L Requirements

Chemical Compound	Percent by Volume
a. Ethylbenzene and toluene	20 max
b. Aromatic compounds with eight or more carbon atoms except ethylbenzene	8 max
c. Solvents with an olefinic or cyclo-olefinic type of unsaturation	Negative Test
d. Ketones	Negative Test
e. Total of a + b	20 Max

Composition L limits the aromatic solvent content — measured by gas-liquid chromatography — and prohibits the presence of ketones. This latter restriction is necessarily more stringent than Rule 442 (Table 2) because of the previous lack of a satisfactory method for the determination of branched chain ketones.

Table 2. California Rule 442 Requirements

Chemical Compound	Percent by Volume
a. A combination of ethylbenzene, toluene, trichloroethylene, and ketones having branched hydrocarbon structures	20 max
b. A combination of aromatic compounds (except ethylbenzene) with 8 or more carbon atoms to the molecule	8 max
c. A combination of compounds having an olefinic or cyclo-olefinic type of unsaturation	5 max
d. Total of a + b + c	20 max

Reported methods for the determination of ketones have utilized widely varying approaches, but the more effective procedures have involved gas-liquid chromatography with sample pretreatment. The pretreatment methods have included ketone extraction with semicarbazide,¹ sodium metabisulfite,² and amino alkylpolysiloxanes.³ These extraction methods identify ketones by chromatogram peak elimination; however, interpretation and quantitation are sometimes difficult for complex solvents.

Another approach was the isolation of the ketones by formation of their 2,4-dinitrophenylhydrazones and their regeneration through an exchange reaction with α -ketoglutaric acid.⁴ This method was demonstrated for only diethyl ketone and lower molecular weight ketones.

A method is presented in this report for the determination of ketones from coating solvents where the ketones are isolated as their 2,4-dinitrophenylhydrazones and then regenerated with levulinic acid containing 10 percent (V/V) of one normal hydrochloric acid. After an alkaline treatment to remove the levulinic acid and hydrochloric acid, quantitation is made with gas-liquid chromatography. The ketones are identified from previously determined retention times.

¹ D. A. Cronin, *Journal of Chromatography*, 64, 25 (1972).

² J. K. Haken et al, *Journal of Chromatographic Science*, 10, 566 (1972).

³ J. R. Ashes and J. K. Haken, *Analytical Chemistry*, 45, 1131 (1973).

⁴ H. Halvarson, *Journal of Chromatography*, 57, 406 (1971).

II. DETAILS OF TEST

3. **Qualitative Tests for Ketones.** The solvent obtained from distillation method 7355 of Federal Test Method Standard No. 141 is subjected to two qualitative tests for ketones.

Qualitative Test No. 1. Pipet 0.3 ml of the solvent into a micro test tube. Add 2 drops of glacial acetic acid followed by 2 drops of saturated sodium nitroprusside in 50:30:20 – water:isopropyl alcohol:ethyl alcohol. After thorough mixing, tilt the test tube to an angle of 30 degrees and add 5 drops of 28-percent ammonium hydroxide. Mix the solution and allow the color to develop for one minute. An orange-to-purple color is positive. If the color does not develop, the following test is applied.

Qualitative Test No. 2. Place 1 drop of the solvent into a micro test tube to which has been added 2 mg of p-nitrophenylhydrazine dissolved in 10 ml of isopropyl alcohol and 5 drops of concentrated hydrochloric acid. Boil the mixture for 30 seconds and add 3 drops of a saturated solution of potassium hydroxide in 95:1 – isopropyl alcohol:water. An orange-to-red-violet color indicates the presence of ketones. When either of the above qualitative tests is positive, the sample is analyzed further as follows.

4. Isolation and Regeneration of Ketones from Coating Solvents.

a. **Preparation of Reagent.**⁵ Place 6.0-6.1 g of 2,4-dinitrophenylhydrazine (DNPH) into a 125-ml Erlenmeyer flask. Slowly and carefully add 30 ml of concentrated sulfuric acid while stirring and cooling in an ice bath. Stir about 1 hour to dissolve the DNPH. Let cool to room temperature then carefully add 90 ml of a 7:2 mixture of 95 percent ethanol:water in 1- to 2-ml increments. Cool in an ice bath for about 10 minutes. Crystals may form. Transfer the reagent to a 200-ml Erlenmeyer flask with the aid of another 90 ml of the 7:2 95 percent ethyl alcohol:water mixture, adding it rapidly and with agitation. Cover the flask with an acid-resistant closure and allow to stand overnight. Transfer the reagent to a glass-stoppered bottle, filtering through glass wool if a residue is present.

b. **Procedure.** Pipet 1.0 ml of the solvent into a 50-ml glass stoppered Erlenmeyer flask containing a Teflon-coated magnetic stirring bar. Add 25 ml of the DNPH reagent and stopper the flask. (This procedure is designed for solvent mixtures containing 5 percent or less ketones. If more are found to be present, the original

⁵ R. L. Shriner, R. C. Fuson, and D. Y. Curtin, *The Systematic Identification of Organic Compounds*, 5th Ed., p. 126.

solvent blend should be quantitatively thinned with mineral spirits and an aliquot of this dilution carried through the procedure.) Stir for 1 hour after the initial appearance of a precipitate. Place the flask in a refrigerator and swirl periodically over a period of 100 minutes. Vacuum filter by transferring the precipitate and stirring bar to a 15-ml medium frit glass crucible using 25 ml of 1 N hydrochloric acid followed by 25 ml of water for the transfer and washing. Maintain the vacuum overnight. More crystals will form in the filtrate. Refilter through the same crucible using a minimum amount of water for transferring and washing. Maintain the vacuum for about 1 hour. Transfer the crucible containing the precipitate and stirring bar to a one-necked, 50-ml, resin-reaction flask. Add 40 ml of levulinic acid which contains 10 percent of 1 N hydrochloric acid by volume. Clamp the top, which is fitted with a Graham reflux condenser, in place and loosely stopper the top of the condenser. Warm on a steam bath for 80 minutes with periodic agitation. Cool the mixture to room temperature and quantitatively transfer it to a glass stoppered container using ethyl ether. The ether solution of the extracted ketones is ready for the following treatment and chromatographic analysis.

5. Addition of Internal Standard and Removal of Levulinic Acid. Add 0.1 g of nonane (internal standard) weighted to the nearest milligram to the ether solution of the extracted ketones. Mix thoroughly. Transfer 2 ml of the above mixture to a 5-inch by 1/2-inch test tube and add 1 ml of distilled water. Stopper the test tube, agitate gently, and cool to equilibrium in an ice-water bath. Add, dropwise, 1 ml of 40-percent aqueous sodium hydroxide. Maintain the temperature below 20°C and agitate intermittently for 5 minutes. Centrifuge the mixture until the two layers separate completely. Transfer most of the upper layer to a 2-ml, septum-fitted vial and reserve for chromatographic analysis.

6. Gas-Liquid Chromatographic Analysis. A gas chromatograph equipped with a flame-ionization detector is used to determine the ketone concentrations. The chromatographic column consists of a 3-foot by 1/8-inch Teflon-coated, stainless steel tube filled with Porapak Q. With a syringe, inject 2 μ l of the solution prepared as above into the gas chromatograph which is operated under the following conditions:

Carrier (helium) flow-rate	25 ml/min
Injection port temperature	250 °C
Flame ionization detector temperature	300 °C
Initial column temperature	180 °C
Rate of column temperature increase beginning at 10 min.	3 °C/min

Final column temperature	225 °C
Total length of run	40 min.

Identify the ketones from previously determined retention times.

Calculate the concentration of each ketone from the relationship:

$$C_x = C_{IS} \times \frac{A_x}{A_{IS}} \times F$$

where: C_x is the ketone concentration.
 C_{IS} is the internal standard concentration.
 A_x is the area under the ketone peak
 A_{IS} is the area under the internal standard peak.
 F is a correction factor determined with known concentrations.

7. **Results.** The two qualitative tests (paragraph 3) were adopted as a means for determining the presence of 4 frequently used ketones. The first described test, a modification of the Lange spot test for ketones, will detect the following three ketones: methyl isobutyl ketone, methyl isoamyl ketone, and mesityl oxide in concentrations as low as 1 percent in mineral spirits. Mineral spirits alone yields an off-yellow color compared to an orange to purple color when one of these three ketones is present.

The presence of isophorone can be detected by Qualitative Test Number 2, which is applied to unknown samples when no color develops with Qualitative Test Number 1. This modified Feigl test⁶ is satisfactory only for isophorone and is effective for concentrations as low as 2 percent. The color changes from orange to purple as the isophorone concentration is increased from 2 to 5 percent. Mineral spirits containing no ketone yields a bright yellow color when subjected to the second qualitative test.

A number of well-known reactions suggest themselves as possible means for separation of ketones from the other solvent materials. Among these are oxime formation, Schiff base formation, bisulfite addition, hydrazone formation, and formation of Girard-type adducts.

Since Girard reagents combine with ketonic substances to yield water soluble hydrazones, such an approach would facilitate the separation from both non-ketonic and water insoluble compounds, including a major constituent of many coatings solvents, mineral spirits. For this and the following reasons, Girard-type reagents were chosen for initial investigation:

⁶ F. Feigl, V. Anger, and G. Fischer, *Microchim. Acta*, 878 (1962).

- a. Reported ease of reaction.
- b. Reported ease of regeneration.
- c. Broad scope of reported usage.
- d. Use in quantitative procedures.
- e. Use in difficult separations from complex mixtures.

Girard Reagent T was tested in 5-percent (V/V) blends of individual ketones in an exempt ketone-free mineral spirits solvent to determine the efficiency of isolation and recovery, with the recovered materials being analyzed by gas-liquid chromatography.

A rather intense effort to develop a procedure which was satisfactory for the ketones of interest was made, including some exploratory efforts using Girard Reagent P. Since both the percent recovery and precision of these determinations for the majority of the ketones examined were unacceptable, it was decided to investigate another approach.

Development of isolation and regeneration procedure. The reagent 2,4-dinitrophenylhydrazine is used for qualitative determinations for carbonyl compounds. As previously stated, this reagent has also been used in some instances for the quantitative determination of some specific compounds of this class.

The following three formulations were chosen for testing:

- a. DNPH in acidic aqueous alcoholic solution (ca. 28 mg/ml).⁷
- b. DNPH in dimethyl sulfoxide, 20 percent solution.⁸
- c. DNPH in diglyme (34 mg/ml).⁹

The first formulation was found to be the most satisfactory and, therefore, was chosen for further investigation.

⁷ R. L. Shriner, R. C. Fuson, and D. Y. Curtin, *The Systematic Identification of Organic Compounds*, 5th Ed., p. 126.

⁸ J. Parrick and J. W. Rasburn, *Canadian Journal of Chemistry*, 43, 3453 (1965).

⁹ H. J. Shine, *Journal of Organic Chemistry*, 24, 252 (1959).

For the regeneration of the free ketones, the procedure reported by Keeney¹⁰ was examined and found to be suitable.

Figure 1 illustrates the chromatogram of an ethyl ether solution of the four ketones selected for this study. The solution also contained nonane as the internal standard. Relative retention times determined from this figure and calculated with nonane as 1.0 are shown in Table 3.

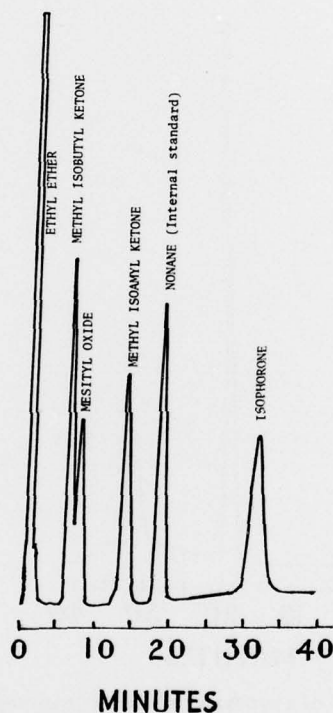


Figure 1. Separation of ether solution of selected ketones and internal standard.

Table 3. Relative Retention Times Calculated with Nonane
(Internal Standard) as 1.0

Ketone	Relative Retention Time
Methyl isobutyl ketone	0.33
Mesityl oxide	0.41
Methyl isoamyl ketone	0.75
Isophorone	1.70

¹⁰ M. Keeney, *Analytical Chemistry*, 29, 1489 (1957).

Figure 2 shows a chromatogram of a 5-percent isophorone solution in mineral spirits after having been processed by the developed method. Similar determinations were made for each of the selected ketones, and recovery values (Table 4) were determined from the chromatograms. Table 5 indicates the high reproducibility of the method.

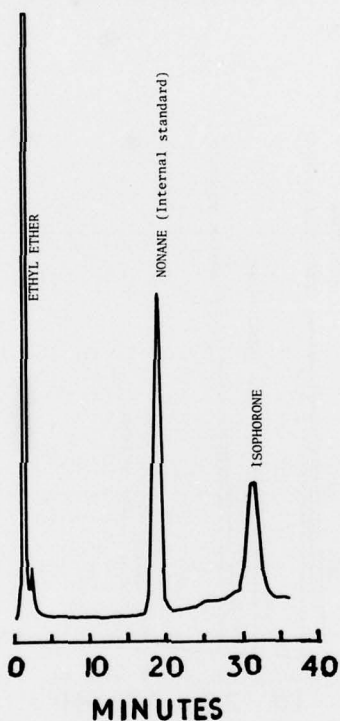


Figure 2. Analysis of a synthetic mixture containing isophorone.

Table 4. Determination of Ketone Recovery Values

Ketone	Percent Recovery		
	Run 1	Run 2	Run 3
Methyl isobutyl ketone	83.6	85.1	86.0
Methyl isoamyl ketone	85.8	88.0	84.5
Mesityl oxide	63.0	62.9	62.1
Isophorone	91.3	88.0	91.6

Table 5. Determination of Method Reproducibility

Ketone	Percent Recovery		
	Analyst 1	Analyst 2	Analyst 3
Methyl isobutyl ketone	85	87	*
Methyl isoamyl ketone	86	87.5	*
Mesityl oxide	63	*	61
Isophorone	90	89	*

* Not performed.

Correction factors (F) calculated from the Table 4 averages of the percent recoveries are shown in Table 6.

Table 6. Correction Factors Calculated from the Averages of Percent Recovery

Ketone	Average Percent Recovery	F
		$\frac{100}{\text{Percent Recovery}}$
Methyl isobutyl ketone	84.9	1.2
Methyl isoamyl ketone	86.1	1.2
Mesityl oxide	62.7	1.6
Isophorone	90.3	1.1

Using these correction factors, the quantitative procedures were further evaluated by running mixtures of the 4 ketones, 2 at the 5-percent level and 2 at the 2.5-percent level. The results are shown in Table 7. Figure 3 illustrates the chromatogram obtained for mixture 1.

Table 7. Test Results from Known Mixtures

Ketone	Mixture 1		Mixture 2		Mixture 3		Mixture 4	
	Percent Present	Percent Found	Percent Present	Percent Found	Percent Present	Percent Found	Percent Present	Percent Found
Methyl isobutyl ketone	5.0	5.1	5.0	5.0	2.5	2.5	2.5	2.4
Methyl isoamyl ketone	5.0	4.8	5.0	4.9	2.5	2.4	2.5	2.4
Mesityl oxide	5.0	5.1	5.0	5.2	2.5	2.4	2.5	2.4
Isophorone	5.0	5.0	5.0	5.0	2.5	2.4	2.5	2.6

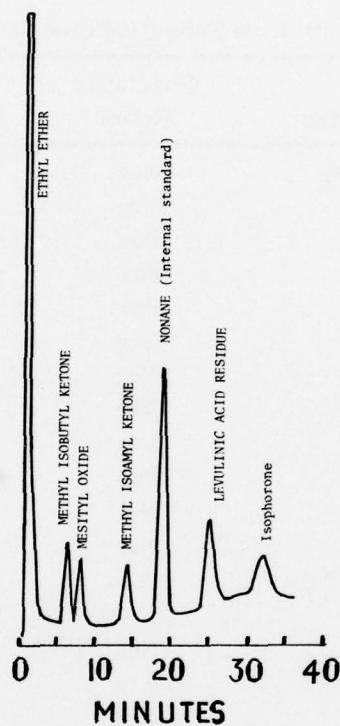


Figure 3. Analysis of a synthetic mixture containing four ketones.

The two qualitative tests followed by the two-step quantitative procedure in cases of positives were applied to a random sampling of solvents which have been obtained by distillation from Composition L, QPL samples over the past 4 years. The qualitative results are given in Table 8 and show a high degree of conformity with the present no-ketone requirement. Quantitative ketone results are also shown in Table 8 along with the original aromatic (toluene plus ethylbenzene) results to check conformity with Rule 442 (Table 2). Analysis of the T240 sample (Table 8) is shown in Figure 4.

Table 8. Test Results from Submitted Composition L Samples

Lab ID No.	Specification	Qualitative Result	Percent Ketones	Percent Aromatic
TB60	MIL-E-52798	Neg		
TB65	" "	Neg		
TB72	" "	Pos	<1	5
TB89	" "	Pos	<1	6
TB101	" "	Pos	<1	7
TB106	" "	Neg		
TB108	" "	Neg		
TB109	" "	Neg		
TB122	" "	Neg		
TB97	" "	Pos	<1	10
TB110	" "	Neg		
TB119	" "	Neg		
T240	MIL-P-52192 Part B	Pos	<1 MEK* + 5 M1BK	6
T032	TT-489	Neg		
T202	"	Neg		
T211	"	Neg		
T256	"	Pos	<1	8
T281	"	Neg		
T288	"	Neg		
T294	"	Neg		
T283	TT-E-516	Neg		
T286	"	Neg		
T287	"	Neg		
T232	MIL-E-46096	Neg		
T285	" "	Neg		
TB124	MIL-E-52891	Neg		

* Methyl ethyl ketone.

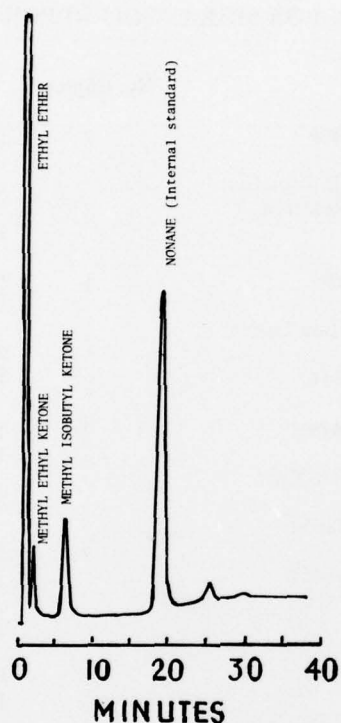


Figure 4. Analysis of unknown sample: Laboratory ID T240.

III. CONCLUSION

8. **Conclusion.** A procedure has been developed for the determination of the more commonly used branched chain ketones in organic coatings. The procedure consists of two qualitative tests for preliminary identification of solvents containing ketones, an isolation and regeneration method, and finally, quantitation by gas-liquid chromatography. Ketone identification is made by retention time on the chromatogram.

Future plans include the incorporation of these methods into new Military Specifications that require the Composition L solvent. Future plans also cover the inclusion of the procedure in revisions of current specifications by amendment as is deemed necessary. At the same time, Composition L requirements will be liberalized to more closely match Rule 442.

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